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(54) **Catalyst composition with high efficiency for the production of light olefins**

(57) The present invention is directed to a catalyst composition for FCC which has high efficiency in the production of light olefins while maintaining the bottoms conversion. Said catalyst composition is prepared by:

a) ex situ activating an olefin selective zeolite with at least 10 wt% of a phosphorus-containing compound, calculated as P_2O_5 based on the total amount of olefin selective zeolite.

b) combining the activated olefin selective zeolite with 10-40 wt % catalytic cracking component, binder and 0-25 wt% silica in a slurry so that the total amount of amorphous alumina present in the final catalyst composition is at least 10 wt%, and
c) spray-drying the slurry to form catalyst particles.

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Description

[0001] The present invention is directed to a catalyst composition for FCC which has high efficiency in the production of C₃ and C₅ olefins, i.e. light olefins. In FCC practice, there are two ways to increase light olefin selectivity.

[0002] The first is to increase the reaction temperature. This will increase the contribution of thermal cracking, which leads to increased formation of lighter products. For instance, in the so-called DCC (Deep Catalytic Cracking) process, a specific type of the FCC process, higher temperatures and increased amounts of steam are used. However, thermal cracking is not very selective and produces large amounts of relatively invaluable products like hydrogen, methane, ethane, and ethylene in the "wet gas" (which contains H₂ and C₁-C₄ products). Wet gas compression often limits refinery operation.

[0003] The second solution is to add a olefin selective zeolite-containing additive such as a ZSM-5-containing additive. Conventional additives usually contain phosphorus-activated ZSM-5, which selectively converts primary cracking products (e.g., gasoline olefins) to C₃ and C₄ olefins. Improvement of the activity or the selectivity with phosphorus is known to increase the effectiveness of ZSM-5. For instance, EP-A- 511 013 describes the treatment of ZSM-5 with phosphorus to increase the propylene selectivity. Further, US 5,472,594 describes a process for converting a hydrocarbon feed to a product containing improved yields of C₄/C₅ olefins with a catalyst composition containing zeolite Y and an additive comprising a phosphorus-containing medium pore zeolite such as ZSM-5. Also Mobil's WO 98/41595 describes a process for the catalytic cracking of a hydrocarbon feedstock to produce an enhanced yield of C₃ to C₅ olefins using a catalyst composition comprising a large pore molecular sieve such as zeolite Y and an additive comprising a phosphorus-containing ZSM-5 blended in with the base catalyst containing zeolite Y. The same is described in US 5,456,821. WO 94/13754 describes the same process using a catalyst composition containing a large pore molecular sieve and an additive containing a specific ZSM-5 which optionally contains 1.5 to 5.5 wt% elemental phosphorus. Also US 5,521,133 describes the preparation of a ZSM-5 additive by injecting a ZSM-5 and kaolin slurry with phosphoric acid prior to spray-drying.

[0004] Additives, however, dilute the catalyst inventory and will decrease bottoms conversion. In the past, it was tried to incorporate phosphorus-activated ZSM-5 as a component into FCC catalyst compositions. In US 5,110,776 a phosphorus treatment was used to improve the attrition resistance of catalyst compositions. Here a Y zeolite is treated with a phosphorus-containing aqueous solution, and said treated zeolite is directly combined with a matrix precursor to form a slurry. Said slurry is spray-dried. The matrix precursor used comprises up to 3.4 wt% alumina, about 25 wt% clay, and about 45 wt% silica. Although it is mentioned that ZSM-5 may be used, all of the examples are directed to the use of zeolite Y. The above-described US 5,472,594 mentions mixing of the phosphorus-activated ZSM-5 with a matrix and Y zeolite and spray-drying to form an FCC catalyst, but gives no indication of how this can be done, nor of the type of matrix to be used. The examples only describe ZSM-5 additives which are mechanically mixed with base catalysts. Mobil's 5,126,298 and US 5,231,064 describe the preparation of a catalyst composition by providing a zeolite slurry and two types of clay slurries, treating at least one of these slurries with a source of phosphorus, combining the slurries, and spray-drying at a pH of below 3. The catalyst matrix described here does not contain any added silica and/or alumina. As will be explained below, phosphorus-activated olefin selective zeolites such as ZSM-5 can only be incorporated into a catalyst composition as a component when specific measures are taken.

[0005] Conventional FCC catalyst compositions which are also suitable for bottoms cracking contain a catalytic cracking component and amorphous alumina. Catalytic cracking components are either crystalline such as zeolite Y and zeolite X or amorphous such as silica-alumina. Amorphous alumina is necessary to provide the bottoms conversion. Amorphous alumina may also be used as a binder to provide the matrix with enough binding function to properly bind the crystalline cracking component when present. Thus, said amorphous alumina is either present in the matrix, i.e., an active matrix is used, or in the amorphous cracking component in the form of silica-alumina. In other words, a catalyst composition with good bottoms conversion contains at least 10 wt% amorphous alumina. With the term amorphous alumina an alumina is meant which comprises bottoms cracking activity. This means that some crystallinity may be present. It was found that when the phosphorus compound used for activation of the olefin selective zeolite is added to the catalyst strike, i.e., the spray-drying slurry containing the matrix components and the catalytic cracking component, it interferes with the amorphous alumina present in the strike. As a result at present no catalyst compositions are on the market which effectively combine olefin selective zeolites with high amounts of alumina either in the matrix or in the amorphous cracking component. The object of the invention is to provide a catalyst composition with a high light olefin selectivity while maintaining the bottoms conversion.

[0006] To this end the olefin selective zeolite is *ex situ* activated, before it is added to a conventional FCC catalyst formulation. *Ex situ* activation of olefin selective zeolite is achieved by treating the zeolite with a phosphate source, followed by drying and calcination. Subsequently the activated olefin selective zeolite is added to the catalyst matrix. It was found that the *ex situ* activation of olefin selective zeolite immobilizes the phosphate enough to avoid its interference with the catalyst matrix or catalytic cracking component after addition thereto. This allows the olefin selective zeolite to exert its selective cracking on the primary products, while not diluting the inventory. Thus, the refiner can

actually lower the riser top temperature, which will allow a lower "wet gas" make, while also the contribution of the Y-zeolite to the C₃- and C₄-selectivity will be lowered. At the same time, the bottoms conversion will be maintained, because the matrix activity can be tailored to the formulation. It must be noted that Sinopec's EP-A1-0909-582 describes *ex situ* phosphorus activation. It describes the preparation of a catalyst composition for the production of light olefins by mixing the matrix components, adding a mixture of a Y-type zeolite and a high silica zeolite with pentasil structure such as ZSM-5 containing 2-8 wt% (based on the weight of the zeolite) of phosphorus (calculated as P₂O₅) and 0.3-3 wt% of aluminium or magnesium or calcium. The zeolite mixture is treated with an aqueous solution of phosphorus/aluminium or phosphorus/magnesium or calcium compounds, dried, and calcined prior to the addition to the matrix components. The amounts of phosphorus used here are not sufficient to obtain a high light olefin yield. NL-9301333 describes the *ex situ* activation of ZRP zeolite with an aluminium phosphate sol. Said activated ZRP zeolite is incorporated into a catalyst composition containing zeolite Y, clay, and organic oxides.

[0007] The present invention is directed to a process for the preparation of a catalyst composition comprising the following steps:

- a) *ex situ* activating an olefin selective zeolite with at least 10 wt% of a phosphorus-containing compound, calculated as P₂O₅ based on the total amount of olefin selective zeolite,
- b) combining the activated olefin selective zeolite with 10-40 wt % catalytic cracking component, binder, and 0-25 wt% silica in a slurry so that the total amount of amorphous alumina in the final catalyst composition is at least 10 wt%, and
- c) spray-drying the slurry to form catalyst particles.

[0008] As was mentioned above, the *ex situ* activation comprises contacting the olefin selective zeolite with a phosphorus-containing compound in solution or liquid. A suitable phosphorus-containing compound, i.e. any phosphorus-containing compound having a covalent or ionic constituent capable of reacting with hydrogen ion, may be employed, for example phosphoric acid and its salts such as ammonium dihydrogen phosphate and diammonium hydrogen phosphate, ammonium hypophosphate, ammonium orthophosphate, ammonium dihydrogen orthophosphate, ammonium hydrogen orthophosphate, triammonium phosphate, phosphines, and phosphites. Suitable phosphorus-containing compounds include derivatives of groups represented by PX₃, RPX₂, R₂PX, R₁P, R₃P=O, RPO₂, RPO(OX)₂, PO(OX)₃, R₂P(O)OX, RP(OX)₂, ROP(OX)₂, and (RO)₂POP(OR)₂, wherein R is an alkyl or phenyl radical and X is hydrogen, R or halide. These compounds include primary, RPH₂, secondary, R₂PH, and tertiary, R₃P, phosphines such as butyl phosphine; tertiary phosphine oxides, R₃PO, such as tributylphosphine; primary, RP(O)(OX)₂, and secondary, R₂P(O)OX, phosphonic acids such as benzene phosphonic acid; esters of the phosphonic acids such as diethyl phosphonate, (RO)₂P(O)H, dialkyl phosphinates, (RO)P(O)R₂; phosphinous acids, R₂POX, such as diethyl-phosphinous acid, primary, (RO)P(OX)₂, secondary, (RO)₂POX, and tertiary, (RO)₃P, phosphites; and esters thereof such as monopropyl ester, alkylalkyl phosphinites, (RO)P₂, and dialkyl phosphonite, (RO)₂PR esters. Examples of phosphite esters include trimethyl phosphite, triethyl phosphite, diisopropyl phosphite, butyl phosphite; and pyrophosphites such as tetrapyrophosphite. The alkyl groups in the mentioned compounds contain 1 to 4 carbon atoms. Other suitable phosphorus-containing compounds include phosphorus halides such as phosphorus trichloride, bromide, and iodide, alkyl phosphoro-dichloridites, (RO)PCl₂, dialkyl phosphorochloridites, (RO)₂PCl, alkyl phosphonochloridates, (RO)(R)P(O)Cl, and dialkyl phosphinochloridates, R₂P(O)Cl.

The reaction of the olefin selective zeolite with the phosphorus-containing compound is effected by contact. Where the phosphorus-containing compound is a liquid, said compound can be used *per se* or in solution in a solvent. If a solid compound is used, it must be solved so as to ensure contact with the olefin selective zeolite.

[0009] It is preferred to use phosphorus-containing compounds which do not leave undesirable metals on the olefin selective zeolite. Therefore, the addition of sodium, aluminium, calcium, and magnesium salts is not advised. If phosphorus-containing compounds are used which do not contain metal ions which are undesired on olefin selective zeolites, such as ammonium dihydrogen phosphate and diammonium hydrogen phosphate, ammonium hypophosphate, ammonium orthophosphate, ammonium dihydrogen orthophosphate, ammonium hydrogen orthophosphate, triammonium phosphate, and organic phosphates, phosphines, and phosphates, there is no need to filter the phosphate-containing compound/Olefin selective zeolite mixture prior to the drying step.

[0010] Olefin selective zeolites are defined as zeolites having a silica/alumina ratio above 10, preferably above 15 and upto 12 rings. Examples of suitable olefin selective zeolites are MFI-type zeolites, MEL-type zeolites such as ZSM-11, ZSM-12, MTW-type zeolites such as ZSM-12, MWW-type zeolites such as MCM-22, MCM-36, MCM-49, MCM-56, and BEA-type zeolites such as zeolite beta. MFI-type zeolites are preferred.

[0011] MFI-type zeolites are as defined in the *ATLAS OF ZEOLITE STRUCTURE TYPES*, W.M. Meier and D.H. Olson, 3rd revised edition (1992), Butterworth-Heinemann, and include ZSM-5, ST-5, ZSM-8, ZSM-11, silicalite, LZ-105, LZ-222, LZ-223, LZ-241, LZ-269, LZ-242, AMS-1B, AZ-1, BOR-C, Boralite, Encilite, FZ-1, NU-4, NU-5, T5-1, TSZ, TSZ-III, TZ01, TZ, USC-4, USI-108, ZBH, ZB-11, ZBM-30, ZKQ-1B, ZMQ-TB. It should be noted that the ZRP zeolite

as described in NL 9301333 is not considered a MFI-type zeolite within the context of this description.

[0012] After treatment with the phosphorus-containing compound, the treated olefin selective zeolite is dried and subsequently calcined at a temperature between 300 and 1000°C, preferably between 450 and 700°C, for about 15 minutes to 24 hours.

[0013] It was found that the olefin selective zeolite should be treated with at least 10 wt% of the phosphorus-containing compound, calculated as P_2O_5 based on the total amount of olefin selective zeolite, to ensure proper light olefin selectivity, but it is preferred to use at least 12 wt% of the phosphorus-containing compound, calculated as P_2O_5 based on the total amount of olefin selective zeolite.

[0014] In general, it is preferred to adjust the pH of the olefin selective zeolite/phosphorus-containing compound mixture to 4.5 or above, for instance with ammonia, before drying in order to avoid any undesirable acid attack of the zeolite or active matrix.

[0015] After *ex situ* activation, the activated olefin selective zeolite is combined with the catalytic cracking component, binder, and silica. Catalytic cracking components are either crystalline such as zeolite Y and zeolite X or amorphous such as silica-alumina. Suitable zeolites Y and zeolite X are all zeolites Y and zeolites X which are normally used in FCC catalyst compositions and may be in the hydrogen form, the ammonium form, or in ion exchanged form, e.g. one or more rare earth metals. Examples are dealuminized zeolite Y, silica-enriched dealuminated zeolite Y, and ultrastable zeolite Y (USY). The zeolite Y and zeolite X may also be treated with a phosphorus-containing compound. Also combinations of catalytic cracking components may be used.

[0016] As mentioned above, the amount of amorphous alumina in the final catalyst composition should be at least 10 wt% so as to ensure sufficient bottoms cracking activity. When silica-alumina is present as the catalytic cracking component, the alumina present herein provides bottoms cracking. In that case, any binder may be used, as long as the total amount of amorphous alumina in the final catalyst composition is at least 10 wt%. Suitable binders are alumina binders, alumina-silica binders or silica binders. Said binders are all aluminas, silicas and silica-aluminas which are normally applied in FCC catalyst compositions. The alumina binder may be (pseudo)boehmite or alumina sol. Usually, the alumina binder is peptized before or during its combination with the other catalyst composition components. Further, silica and/or crystalline alumina may be present in the strike and optionally fillers such as clay, for instance kaolin, are used. Usually, up to 20 wt% binder, preferably alumina binder, is present in the final catalyst composition, but also higher amounts may be applied.

[0017] The various catalyst components can be combined in any sequence, but it is preferred to add the phosphorus treated olefin selective zeolite to the strike last, so as to avoid any interference of the phosphorus with the amorphous alumina. It is further preferred to add the phosphorus treated olefin selective zeolite separately from the zeolite Y, so as to avoid any interference with the catalytic cracking component.

[0018] It is preferred to use zeolite Y as the catalytic cracking component, because this provides extremely suitable catalyst compositions.

[0019] As the catalyst composition resulting from the above-described process appears to have improved properties compared with known catalyst compositions, the present invention is also directed to a catalyst composition obtainable by the process of the invention. To this end the present invention is directed to a catalyst composition comprising:

- a) 10 to 40 wt % catalytic cracking component,
- b) 0.1 to 85 wt % olefin selective zeolite treated *ex situ* with phosphorus, wherein the olefin selective zeolite is treated with at least 10 wt% phosphorus-containing compound, calculated as P_2O_5 based on the total amount of olefin selective zeolite,
- c) binder,
- d) 0-25 wt % silica.

wherein the total amount of amorphous alumina in the final catalyst composition is at least 10 wt%.

[0020] The invention is further directed to a catalyst composition wherein the olefin selective zeolite is treated with at least 12 wt% phosphorus-containing compound, calculated as P_2O_5 based on the total amount of olefin selective zeolite.

[0021] Said catalyst composition can suitably be used in the catalytic cracking of hydrocarbon feedstocks and has high efficiency in the production of light olefins while maintaining the bottoms conversion. The catalyst composition may also be used in the so-called DCC process even when using lower temperatures than usual in DCC processes.

[0022] The present invention is further illustrated by the Examples which should in no way be construed as being limitative.

EXAMPLES

Example 1: Ex situ activation of ZSM-5

[0023] ZSM-5 with a SAR of 25 was slurried to obtain a 30 % slurry. H_3PO_4 was added to levels of 2, 6, 8, 10, 15 and 20 wt% P_2O_5 on ZSM-5, respectively. The pH was adjusted to above 4.5 with ammonia. The mixture was dried at 120 °C overnight and calcined at 600 °C for 1 hour. 5 wt% ex situ activated ZSM-5 was added as the last component to a strike containing 13.5 wt% REY zeolite, 18 wt% peptized pseudoboehmite and 5 wt% silica. The strike was spray dried.

[0024] The catalyst compositions were steamed for 20 hours at 788 °C. The physical properties of the catalyst compositions are listed in TABLE 1.

Example 2: Calcination at different temperatures

[0025] Example 1 was repeated using H_3PO_4 levels of 2 and 6 wt% P_2O_5 on ZSM-5, respectively. Now, the calcination temperature was 400 °C, with the other conditions being the same as in example 1. The physical properties of the resulting catalyst composition appeared to be approximately the same as the catalyst composition obtained with calcining at 600 °C.

Comparative example 3: Additive with base catalyst

[0026] For comparison a base catalyst was prepared with the formulation 15 wt% REY, 20 wt% peptized pseudo-boehmite, and 5 wt% silica which was blended with 10 wt% commercially available ZSM-5 additive containing 25 wt% ZSM-5 [ZCAT HP @ cx Interocat, Eelco is this correct?]. This is the way commercially available additives are usually applied. The catalyst blend was steamed for 20 hours at 788 °C. The physical properties of this comparative catalyst blend is listed in TABLE 1 as ex. 3*.

TABLE 1

Physical properties of catalyst compositions							
	sample 1	sample 2	sample 3	sample 4	sample 5	sample 6	ex. 3*
wt % P_2O_5 added	2	6	8	10	15	20	
Al_2O_3	46.61	46.55	46.34	46.11	44.59	46.31	34.16
SiO_2	48.31	48.05	48.54	48.51	49.57	48.22	59.78
Na_2O	0.16	0.15	0.15	0.23	0.93	0.16	0.16
RE_2O_3	2.15	2.18	2.21	2.22	1.96	2.21	2.22
P_2O_5 (measured)	0.14	0.29	0.39	0.45	0.76	0.71	1.29
PSD	69.67	65.75	-	83.04	73.55	60.25	66.63
PV	0.38	0.38	0.38	0.38	0.37	0.38	0.51
SA_{bet}	198	190	197	191	189	188	194
SA_{bet} (steamed)	114	117	120	123	117	119	114

Example 4: propylene yield.

[0027] The various catalyst compositions were tested using Kuwait vacuum gas oil as a feedstock. The performance at catalyst to oil ratio 4 is reported in TABLE 2. It was acceptable to report at constant catalyst to oil ratio instead of at constant conversion because the activity difference was minimal so that the overall ranking was not changed by this.

TABLE 2

Performance of the catalyst compositions			
	Propylene yield	butylene yield	LPG yield
sample 1 (2% P_2O_5)	7.86	8.02	23.74

TABLE 2 (continued)

Performance of the catalyst compositions			
	Propylene yield	butylene yield	LPG yield
sample 2 (6% P ₂ O ₅)	8.81	8.51	25.35
sample 3 (8% P ₂ O ₅)	8.78	8.40	24.89
sample 4 (10% P ₂ O ₅)	8.82	8.48	24.97
sample 5 (15% P ₂ O ₅)	9.47	8.50	25.98
sample 6 (20% P ₂ O ₅)	9.76	8.73	26.66
Ex 3*	9.10	8.57	25.66

[0028] These results show that with catalyst composition having *ex situ* activated olefin selective zeolite incorporated into the catalyst composition a propylene yield can be obtained comparable with the use of commercially available additives, as long as the Olefin selective zeolite is treated *ex situ* with at least 10 wt% of phosphorus-containing compound. Although with the use of ZSM-5 additives a good propylene yield can be obtained, the bottoms cracking is reduced owing to dilution effects.

Example 5: Ex situ activated ZSM-5-containing catalyst composition

[0029] Another catalyst composition was prepared using the process as described in Example 1 using .. wt% *ex situ* activated ZSM-5 to form a catalyst composition containing .. wt% zeolite y, ..wt% peptized pseudoboehmite, and .. wt% silica. The catalyst composition was steamed for 20 hours at 788 °C.

Comparative example 6: base catalyst

[0030] For comparison a base catalyst was prepared with the same formulation as Example 5 except for the activated ZSM-5. The base catalyst was steamed for 20 hours at 788 °C.

Example 7: propylene yield and bottoms conversion of base catalyst and ZSM-containing catalyst composition

[0031] The catalyst composition of Example 5 and the base catalyst of comparative example 6 were tested using the same feedstock. The performance at catalyst to oil ratio 5 is reported in TABLE 3.

TABLE 3

Performance of a catalyst composition vs. a base catalyst		
	Catalyst composition of Ex. 5	Base catalyst of comparative Ex. 6
Conversion, wt%	73.13	71.07
Propylene yield, wt%	6.21	4.61
bottoms yield, wt%	11.75	14.59

[0032] These results show that with catalyst composition having *ex situ* activated olefin selective zeolite incorporated into the catalyst composition a higher activity can be obtained than with a base catalyst, providing a higher propylene yield and with less bottoms left, i.e. with a higher bottoms cracking.

Claims

1. Process for the preparation of a catalyst composition comprising the following steps:

- ex situ activating a olefin selective zeolite with at least 10 wt% of a phosphorus-containing compound, calculated as P₂O₅ based on the total amount of olefin selective zeolite,
- combining the activated olefin selective zeolite with 10-40 wt % catalytic cracking component, binder and

0-25 wt% silica in a slurry so that the total amount of amorphous alumina present in the final catalyst composition is at least 10 wt%, and
c) spray-drying the slurry to form catalyst particles.

- 5 2. Process for the preparation of a catalyst composition according to claim 1 wherein the olefin selective zeolite is a MFI-type zeolite.
3. Process according to claim 1 or 2 wherein the catalytic cracking component is a zeolite Y.
- 10 4. Process according to any one of claims 1-3 wherein the olefin selective zeolite is treated with at least 12 wt% of a phosphorus-containing compound, calculated as P_2O_5 based on the total amount of olefin selective zeolite.
5. Process according to any one of claims 1-4 wherein the ex situ activation comprises:
15 a) contacting the olefin selective zeolite with a phosphorus -containing compound in solution or liquid,
 b) drying the treated olefin selective zeolite, and
 c) calcining the treated, dried, olefin selective zeolite at a temperature between 300 and 1000 °C.
- 20 6. Process according to claim 5 wherein the pH of the olefin selective zeolite/phosphorus-containing compound mixture is adjusted to 4.5 or above.
7. Catalyst composition obtainable by any one of the processes of claims 1-6.
- 25 8. Catalyst composition comprising:
 a) 10 to 40 wt % catalytic cracking component,
 b) 0.1 to 85 wt % ex situ phosphorus-treated olefin selective zeolite, wherein the olefin selective zeolite is treated with at least 10 wt% phosphorus-containing compound, calculated as P_2O_5 based on the total amount of olefin selective zeolite,
30 c) binder,
 d) 0-25 wt % silica ,

 wherein the total amount of amorphous alumina in the final catalyst composition is at least 10 wt%.
- 35 9. Catalyst composition according to claim 8 wherein the phosphorus-treated olefin selective zeolite is a phosphorus-treated MFI-type zeolite.
10. Catalyst composition according to claim 8 or 9 wherein the cracking component is zeolite Y.
- 40 11. Catalyst composition according to claim 8, 9 or 10 wherein the olefin selective zeolite is treated with at least 12 wt% phosphorus-containing compound, calculated as P_2O_5 based on the total amount of olefin selective zeolite.
- 45 12. Process for the catalytic cracking of a hydrocarbon feedstock wherein a catalyst composition according to any one of claims 7-11 is used.

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EUROPEAN SEARCH REPORT

Application Number
EP 00 20 0092

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The present search report has been drawn up for all claims			
Place of search THE HAGUE		Date of completion of the search 22 June 2000	Examiner De Herdt, O
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**ANNEX TO THE EUROPEAN SEARCH REPORT
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This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report.
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